IN THE DRAWINGS:

See the replacement sheets for Figures 1, 2 and 3.

REMARKS

The Office Action raised issues with regards to the drawings, which are believed to be adequately addressed by the proposed replacement sheets.

The new Abstract of Disclosure is attached hereto.

The specification has been amended consistent with the request to coordinate with the drawings.

Applicant appreciates the Examiner's comments with regards to the 35 U.S.C. §112 issues on the claims. It is believed that the amended claims more than adequately resolve these matters.

Each of the outstanding claims have been rejected under 35 U.S.C. §103 basically over the *Umezawa et al.* (Japanese Laid-Open Application 2002-221479). Attached is a translation of this reference.

As can be seen, the current inventors, Tetsushi Yamaguchi and Makoto Umezawa are the inventors of the cited Japanese application. This application is also assigned to the same assignee, Horiba Ltd., of the present application.

The present application is entitled to a priority date of November 21, 2002 and accordingly the principle reference relied upon, *Umezawa et al.*, Japanese Laid-Open Application 2002-221479 is not prior art to our present invention. Attached is a certified translation of our priority document.

The *Masuda* US Patent No. 6,465,802 was cited for combination with the *Umezawa et al.* reference and was particularly cited for ensuring that the laser light, as it passed through the center of the cell or irradiation region M, would not be deviated by various indices of refraction of sample fluids. Accordingly, one of an outer wall or an inner wall was arranged to be

perpendicular to the incident laser beam while the other wall was relatively inclined to ensure that the path, or optical axis of the laser beam would extend through the middle of the flow cell or the irradiation radiation point M. *Masuda* does not render the current claims anticipated nor obvious.

Claim 3 was rejected over a combination of the *Umezawa et al.* reference in view of the Matsuda reference and further in view of the *Furuya* US Patent No. 5,172,004.

The *Furuya* reference was cited for its use of a shielding plate 6 with a slit positioned between a transparent cell and the scattering light intensity detection section. Basically, the *Furuya* reference teaches, in its description of the prior art, a conventional single photon counting method. In this environment, a single photon counting is utilized for particles having a diameter smaller than one tenth the wavelength of the incident laser beam and the condenser lens is designed to focus an image of the scattered light at a slit 6 so that the "scattered light passes through the slit 6". There is no express teaching in *Furuya* of blocking any undesired noise light that may occur from a transmission through a cell wall. As such, neither the *Masuda* nor the *Furuya* references are capable of providing the claim elements previously allegedly taught by the *Umezawa* reference.

Claim 5 was rejected over a combination of the *Umezawa et al.*, *Masuda* reference, and the *Kubo et al.* US Patent No. 5,696,580.

The *Kubo et al.* reference was cited for disclosing a triangular cell which is shown in Figure 4A for measuring the characteristics of a urine sample. Apparently, a urine sample can have a variation in the concentration or specific gravity and as seen in Figure 4A it is possible to simultaneously measure both the transmission light intensity and a refractive index through use of a triangular cell to act like a prism. See, for example, Column 6, line 45 through Column 7,

line 67. As can be appreciated, this reference is not directed to resolving a noise to signal ratio, but rather is for purposes of detecting a transmitted light intensity and of calculating a refractive index based on the position of a spread measured beam impact along a length of a linear sensor 3 as illustrated in Figure 4A.

Again, the *Kubo et al.* reference does not address the problems recognized and solved by the present invention.

In view of the mooting of the applicability of the *Umezawa et al.* reference as prior art under 35 USC § 103 and further the inadequacies of the secondary references, *Masuda* and *Kubo et al.*, it is believed that the case is now in condition for allowance and early notification of the same is requested.

New Claims 13 and 14 provide an alternative definition of our invention which are also believed allowable.

If the Examiner believes that a telephone interview will help further the prosecution of the case he is respectfully requested to contact the undersigned attorney at the listed phone number.

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I hereby certify that this correspondence is being deposited with the United States Postal Service as First Class Mail in an envelope addressed to Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on September 8, 2006.

By: Sharon Farnus

Sharon Farnus
Signature

Dated: September 8, 2006

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PATENT ABSTRACTS OF JAPAN

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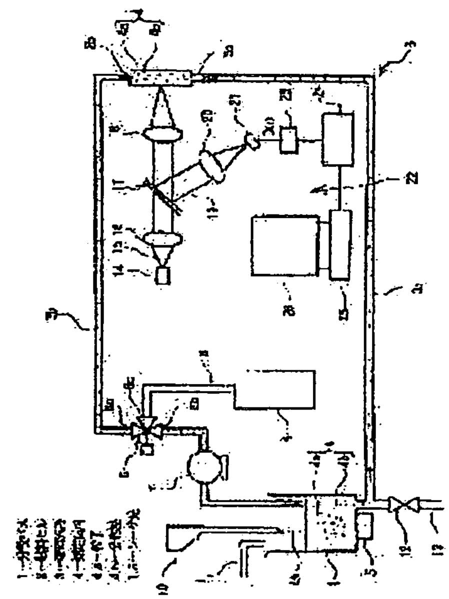
(54) DIAMETER DISTRIBUTION MEASURING APPARATUS FOR DYNAMICALLY LIGHT SCATTERING PARTICLE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a diameter distribution measuring apparatus for dynamically light scattering particle which measures a concentration of a sample used for measuring before measuring a particle diameter distribution, automatically dilutes the sample until the predetermined concentration for measuring is attained, and can efficiently perform the predetermined particle diameter distribution measurement.



SOLUTION: In the diameter distribution measuring apparatus for dynamically light scattering particle in which a measured sample 4 that is a dispersing medium 4b with dispersed particles 4a is accommodated in a sample cell 2 and a laser light 15 irradiates the measured sample 4 and the particle diameter distribution is found based on an intensity distribution in frequency of the light scattered by the particles 4a, the dispersing medium 4b and the dispersed particles 4a are supplied, a dispersing bus 1 for dispersing the dispersed particles 4a in the dispersing medium 4b and the sample cell 2 are connected through a circularly flowing path 3, the measured sample 4 is supplied from the dispersing bus 1 to the sample cell 2 prior to the particle diameter distribution measurement, the laser light 15 irradiates the measured sample 4, the concentration of the measured sample 4 is measured based on an obtained intensity of a scattered light, the measured concentration is compared with a preset target concentration, and a concentration adjustment of the measured sample 4 can be performed based on a result of the comparison.

LEGAL STATUS

[Date of request for examination]

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CLAIMS

[Claim(s)]
[Claim 1] In a sample cell, hold the test portion which distributed the particle in the dispersion medium, and a laser beam is irradiated to this test portion. In the dynamic-light-scattering type particle-size-distribution measuring device which searched for particle size distribution based on the frequency intensity distribution of the light scattered about by said particle While connecting the distributed bus and said sample cell for said dispersion medium and particle being supplied and distributing a particle in a dispersion medium on a circulating flow way In advance of particle-size-distribution measurement, a test portion is supplied from said distributed bus to a sample cell. Irradiate a laser beam to this test portion, and the concentration of said test portion is measured based on the scattered-light reinforcement then obtained. The dynamic-light-scattering type particle-size-distribution measuring device characterized by enabling it to perform concentration adjustment of said test portion based on this comparison result as compared with the target concentration beforehand set up in this measured concentration.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to a dynamic-light-scattering type particle-size-distribution measuring device.
[0002]
[Description of the Prior Art] There are some which use a dynamic-light-scattering type particle-size-distribution measuring device for one of the particle-size-distribution measuring methods. This dynamic-light-scattering type particle-size-distribution measuring device holds the test portion which distributed the particle in the dispersion medium in the sample cell , and irradiates a laser beam to this test portion , particle size distribution is search for based on the frequency intensity distribution of the light scattered about by said particle , and , in addition to a conventional organic pigment , the conventional ceramics , etc. , the needs in a semi-conductor wafer , the abrasive material of a hard disk , and the site of researches and developments of advanced materials , such as an ink jet printer , are increase in recently .
[0003]
[Problem(s) to be Solved by the Invention] By the way, while the so-called batch processing of the above-mentioned dynamic-light-scattering type particle-size-distribution measuring device which supplies a test portion to a sample cell was in use in the former at every measurement, since the measurable concentration of a measuring device needed to be very low, the low-concentration thing or the low-concentration high-concentration thing as a test portion needed to be diluted and it needed to use, the technique of diluting a sample automatically even to the concentration of the measurable range might be adopted. This is considered that there is that cause also in having had much know-how measured by dilution, and many databases in the user using this kind of equipment in the former.
[0004] And it faced diluting said sample, the sample was extracted quantitatively conventionally, and the method of pouring in a diluent quantitatively so that it may be called 100 times or 1000 times to this sample was adopted.
[0005]
[Problem(s) to be Solved by the Invention] However, it was the purpose to carry out dilution adjustment of the test portion at the measurable concentration of a measuring device, and in the sample (particle) which is easy to condense, the above-mentioned technique will be measured while it had been influenced of the condensation by dilution, and also when becoming the measurement in the condition of having differed from the busy condition in the actual site of a particle, there was. And if particle-size-distribution measurement is performed after said condensation has arisen, even if it produces a big change about scattered-light reinforcement or dilutes, scattered-light reinforcement will become feeble, and that predetermined measurement cannot be performed will arise.
[0006] It is offering the dynamic-light-scattering type particle-size-distribution measuring device which enabled it to perform predetermined particle-size-distribution measurement efficiently as this invention was made with careful attention to the above-mentioned matter, that purpose's measured the sample concentration with which measurement is presented before particle-size-

distribution measurement, and diluted a sample automatically until it reaches desired measurement concentration.

[0007]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, in this invention, the test portion which distributed the particle in the dispersion medium is held in a sample cell. In the dynamic-light-scattering type particle-size-distribution measuring device which searched for particle size distribution based on the frequency intensity distribution of the light which irradiated the laser beam to this test portion, and was scattered about by said particle While connecting the distributed bus and said sample cell for said dispersion medium and particle being supplied and distributing a particle in a dispersion medium on a circulating flow way In advance of particle-size-distribution measurement, a test portion is supplied from said distributed bus to a sample cell. A laser beam is irradiated to this test portion, the concentration of said test portion is measured based on the scattered-light reinforcement then obtained, and it enables it to perform concentration adjustment of said test portion based on this comparison result as compared with the target concentration beforehand set up in this measured concentration.

[0008] In the above-mentioned dynamic-light-scattering type particle-size-distribution measuring device, the sample with which measurement is presented can be automatically diluted to concentration (or density range) to dilute. And since concentration can be checked preventing the change of state of the sample by dilution, it is not diluting too much or the lack of dilution is not produced.

[0009]

[Embodiment of the Invention] The gestalt of implementation of invention is explained referring to a drawing. Drawing 1 - drawing 5 show one example of this invention. First, drawing 1 shows roughly an example of the dynamic-light-scattering type particle-size-distribution measuring device of this invention. in this drawing, 1 is a distributed bus, 2 is a sample cell, and both 1 and 2 are connected on the circulating flow way 3.

[0010] It consists of a cylinder-like cistern, and particle 4a which is a measuring object sample, and dispersion-medium 4b which distributes this are mixed, it considers as a test portion 4, the ultrasonic vibrator 5 which vibrates with an oscillator is formed in the bottom outside section, and it is made for said distributed bus 1 to have it prevented that particle 4a condenses in the distributed bus 1.

[0011] Said sample cell 2 consists of the so-called flow cell, sample inlet-port 2a is formed in the end, and sample outlet 2b is formed in the other end.

[0012] Outward trip tubing 3a by which the end was connected to the pars basilaris ossis occipitalis of the distributed bus 1, and the other end was connected to sample inlet-port 2a of a sample cell 2, and an end are connected to sample outlet 2b of a sample cell 2, and said circulating flow way 3 consists of return trip tubing 3b in which the other end carries out opening into the distributed bus 1 in the upper part of the distributed bus 1.

[0013] If the configuration by the side of said distributed bus 1 is explained, the Mikata solenoid valve 6 and the pump 7 for circulation are formed in the downstream of return trip tubing 3b, the 1st and 2nd port 6a and 6b is connected to return trip tubing 3b, respectively, and, as for the Mikata solenoid valve 6 located more in the upstream, the dispersion-medium depot 9 is connected to the 3rd port 6c through tubing 8. And at for example, the time of power-source OFF, Ports 6b and 6c are open for free passage, and at the time of power-source ON, the Mikata solenoid valve 6 is constituted so that Ports 6a and 6b may be open for free passage. Moreover, the particle feed hopper 10 which supplies particle 4a by which weighing capacity was carried out separately in the distributed bus 1, and the dispersant supply pipe 11 which supplies a dispersant in the distributed bus 1 are formed in the upper part of the distributed bus 1. Furthermore, multipoint connection of the drain pipe 13 equipped with the closing motion valve 12 is carried out to the part near the pars basilaris ossis occipitalis of the distributed bus 1 of outward trip tubing 3a.

[0014] In addition, as said dispersion-medium 4b, although liquids, such as water (pure water), ethanol, and an oil, are used, for example, it is properly used suitably according to the class of

particle 4a which is the measuring object. Moreover, make particle 4a easy to distribute in dispersion-medium 4b, and particles, such as a surfactant, are made to repel, for example, said dispersant is supplied by the need.

[0015] And if the configuration by the side of said sample cell 2 is explained, 14 is the laser light source prepared in the 1 side of a sample cell 2, and the lens 16 which collimates the laser beam 15 from a laser light source 14, the beam splitter 17, and the condenser lens 18 are formed in the optical path between this laser light source 14 and sample cell 2 in this order. The hole with which said beam splitter 17 passes a laser beam 15 in the center is prepared. It is prepared so that the reflector may consist of a mirror formed in the sample cell 2-way and the optical axis of a laser beam 15 and the include angle of 45 degrees which emitted the laser light source 14 may be made. It is constituted so that the laser beam 15 which passed said hole may bend the interference light 19 produced by the Doppler shift of the scattered light by particle 4b in a sample cell 2 whenever [proper 90 degrees or less angle] and may reflect. And the lens 20 which condenses this, and the photodetector 21 which changes said interference light 19 into the electric detecting signal D (t) are formed in the optical path of the interference light 19 reflected by the mirror 17.

[0016] moreover, control the signal-processing section 23 which 22 is the signal-processing section, and amplifies the output D of said photodetector 21 (t) as occasion demands, and carries out transform processing to a digital signal, CPU24 which carries out data processing of the detecting signal D (t) outputted from this signal-processing section 23, and searches for particle size distribution, and each part of equipment, or It consists of a personal computer 25 which performs various processings, such as an image processing for displaying the processing result in said CPU24, and processing results, such as particle size distribution, are displayed on a personal computer 25, or the display 26 for displaying various kinds of control screens is attached to it.

[0017] Next, actuation of the dynamic-light-scattering type particle-size-distribution measuring device of the above-mentioned configuration is explained, also referring to drawing 2 - drawing 5 . The dynamic-light-scattering type particle-size-distribution measuring device of this example performs the following actuation automatically according to the program in which it is contained by the personal computer 25.

[0018] (1) First, make the closing motion valve 12 into a closed state, and supply particle 4a and dispersion-medium 4b by which weighing capacity was carried out in the distributed bus 1 (step S1 of drawing 2). When the injection of dispersion-medium 4b carries out time amount ON of the pump 7 suitably in the condition (condition which Ports 6b and 6c are opening for free passage) that the Mikata solenoid valve 6 is off, dispersion-medium 4b of an amount is suitably supplied in the distributed bus 1. At this time, the optimum dose injection of the dispersant may be carried out if needed.

[0019] (2) When the Mikata solenoid valve 6 is turned ON, Kaisei of the circulating flow way 3 is carried out and it turns on a pump 7 by making Ports 6a and 6b open for free passage Particle 4a and dispersion-medium 4b in the distributed bus 1 As it returns to the distributed bus 1 through outward trip tubing 3a, a sample cell 2, return trip tubing 3b, the Mikata solenoid valve 6, and a pump 7, it circulates through the inside of the circulating flow way 3, and it becomes the test portion 4 by which particle 4a distributed in dispersion-medium 4b, and stirring mixing was fully carried out by this (step S2 of drawing 2). In this case, an ultrasonic vibrator 5 is operated and you may make it prevent that particle 4a condenses in the distributed bus 1.

[0020] (3) Turn ON a laser light source 14 and after circulating through said test portion 4 suitably and fully carrying out stirring mixing of this, where a pump 7 is turned OFF, irradiate a laser beam 15 at the test portion 4 in a sample cell 2. That is, the laser beam 15 which came out of the laser light source 14 condenses in a sample cell 2 through a collimate lens 16, a beam splitter 17, and a condenser lens 18. At this time, the laser beams (scattered light) which carried out the Doppler shift by said Brownian motion are scattered about in particle 4a which some laser beams pass through a cell wall side, distributes them in dispersion-medium 4b, and carries out Brownian motion. On the other hand, some laser beams are scattered about in respect of a cell wall (non-scattered light), and the laser beam of the frequency of a basis goes to hard flow.

It interferes each other in said scattered light and non-scattered light, they turn into an interference light, and condense on a photodetector 21 through a condenser lens 18, a beam splitter 17, and a lens 20.
[0021] Said interference light is changed into the electric detecting signal D (t) in a photodetector 21, in the signal-processing section 23, it is processed suitably, and the digitized light-scattering data are incorporated by CPU24. As shown in drawing 3 , said light-scattering data have the amplitude Am according to the concentration of a test portion 4, and can obtain the concentration (measurement concentration) of a test portion 4 based on this amplitude Am (step S3 of drawing 2). In addition, in CPU24, the fast Fourier transform of said light-scattering data is carried out, it asks for a power spectrum, and you may make it search for the particle-size-distribution condition of particle 4a based on this power spectrum.
[0022] And in a personal computer 25, a measurement person in charge sets up target concentration (electrical-potential-difference value) using the screen shown in drawing 4 . Drawing 4 shows an example of the screen in the display 26 when performing automatic concentration adjustment, and the electrical-potential-difference value showing target concentration is 12.50V in this example. And said measurement concentration is measured with target concentration (step S4 of drawing 2). Here, as shown in drawing 5 , when measurement concentration (measurement concentration electrical potential difference) is in agreement with target concentration (target concentration electrical potential difference), it means that adjustment of test portion concentration was completed (step S5 of drawing 2), and shifts to particle-size-distribution measurement. As explained above (3), this particle-size-distribution measurement turns ON a laser light source 14, irradiates a laser beam 15 at the test portion 4 in a sample cell 2, and should just search for particle size distribution based on the frequency intensity distribution of the light scattered about by the particle 4a particle then acquired.
[0023] On the other hand, when said measurement concentration is not in agreement with target concentration, as shown in drawing 2 , it is necessary to perform concentration adjustment of return and a test portion 4 to step S2 further. That is, as shown in the example of a screen of drawing 6 , when larger like 12.50V than target concentration (target concentration electrical potential difference) 10.50V, said measurement concentration (measurement concentration electrical potential difference) sets up a dilution scale factor, and adjusts a test portion 4.
[0024] After opening the closing motion valve 12 about 1 to 2 seconds and specifically discharging a part of test portion 4 in the distributed bus 1, from the dispersion-medium depot 9, dispersion-medium 4b equal to this discharge is supplied to the distributed bus 1, and is diluted. At this time, amount supply of the dispersant may be carried out suitably. Then, let the test portion 4 which consists of particle 4a and dispersion-medium 4b be the test portion 4 by which stirring mixing was fully carried out by circulating the circulating flow way 3 by the technique indicated above (2).
[0025] And as shown in step S3, the concentration of a test portion 4 is measured again.
[0026] Hereafter, the above-mentioned activity is repeated until the concentration of a test portion 4 turns into target concentration. In addition, what is necessary is just to add particle 4a to the distributed bus 1, when the concentration of a test portion 4 is smaller than target concentration. Moreover, as target concentration, some tolerance of ** is prepared, and when measurement concentration enters in the tolerance of this target concentration, it may be made to shift to particle-size-distribution measurement.
[0027] As mentioned above, in the dynamic-light-scattering type particle-size-distribution measuring device of this invention, since it has diluted checking the concentration of a test portion 4 based on scattered-light reinforcement, and checking whether there is any sample concentration within measurable limits in advance of particle-size-distribution measurement, it is not diluting too much or the lack of dilution is not produced. And since particle-size-distribution measurement can be performed after the test portion 4 has become predetermined concentration, the high measurement result of precision can be obtained and predetermined particle-size-distribution measurement can be performed efficiently.
[0028] Since the distributed bus 1 and a sample cell 2 are connected by the circulating flow way 3, the inside of the circulating flow way 3 can be repeated and a test portion 4 can be circulated

especially, while mixed stirring of particle 4a and dispersion-medium 4b is ensured, change of the particle size by condensation and the abrupt change of the scattered-light reinforcement accompanying it can be controlled.
[0029] in addition -- as the dilution approach of a test portion 4 -- concentration -- quantitive -- every [of 2 / a multiple] -- you may make it dilute That is, it acts as the monitor of the amount of the test portion 4 in the distributed bus 1 by the sensor (not shown) formed in the distributed bus 1, moiety wastewater is carried out, two fold serial dilution of the dispersion-medium 4b of this discharge and tales doses is supplied and carried out, and you may make it ask this two fold serial dilution for a dilution scale factor simply from a count.
[0030]
[Effect of the Invention] As explained above, it sets to the dynamic-light-scattering type particle-size-distribution measuring device of this invention. While connecting a distributed bus and a sample cell on a circulating flow way, particle-size-distribution measurement is preceded. Supply a test portion from an account distribution bus to a sample cell, and a laser beam is irradiated to this test portion. Since it enables it to perform concentration adjustment of said test portion based on this comparison result as compared with the target concentration which measures the concentration of said test portion based on the scattered-light reinforcement then obtained, and is beforehand set up in this measured concentration It is efficient even to the concentration which wants to dilute a test portion, and can dilute with uninhabited automatically. And concentration can be checked, preventing the change of state by dilution, therefore desired particle-size-distribution measurement can be performed in the optimal concentration condition, and high measurement of precision can be performed efficiently.

[Translation done.]